Evaluation of Biobased Hydraulic Fluids in Military Construction Equipment

In-Sik Rhee, Ph.D
U.S. Army Tank-Automotive Research, Development, and Engineering Center

ABSTRACT

U.S. Army Tank-Automotive RD&E Center is currently developing biobased hydraulic fluids (BHFs) to replace military industrial and mobility hydraulic fluids that are incompatible with environment. To verify the performances of these biobased fluids in military construction equipments, a joint field demonstration was initiated with US Department of Agricultural (USDA) using ten military construction equipment (i.e., Bulldozer, Scraper, Grader, Loader, Crane, etc.) at Fort Leonard Wood, MO. The field test was successfully completed and the test results showed that BHFs did not provide any abnormal behavior compared to the conventional petroleum based fluids. Based on the test results, this paper will discuss the on-going biobased fluid evaluation program, test results, and findings.

INTRODUCTION

Environmental safety and compliance has recently become the most significant worldwide issue. Over the past decades, many military installations throughout the United States have been contaminated with petroleum and related fuels, lubricants and associated products, such as lubricating oils, greases, hydraulic fluids, aircraft and automotive fuels, and those fuels used for fixed installations[1] . The environmental threats or damage to soils, surface water, and underground water were often caused by leaking containers, accidental spills, or equipment breakdown during active use or storage of these materials. The generation of the potentially hazardous wastes by Petroleum, Oil, and Lubricant products (POL) not only cause both short and long term liability with respect to environmental damage, but can result in deteriorated mission performance and high cleanup costs. Currently, the Resource Conservation and Recovery Act (RCRA)[2] and the DoD Hazardous Waste Minimization (HAZMIN) Policy mandate that all DoD installations must reduce the quantity or volume and toxicity of hazardous waste generated by POL wherever economically practicable and environmentally necessary. To achieve the HAZMIN goals, U.S. Army Tank-Automotive RD&E Center is currently developing the biobased hydraulic fluids (BHFs) to replace military industrial and mobile hydraulic fluids that are incompatible with the environment. Initiating development of such products was most timely in view of the Executive Order No. 13134 on Developing and Promoting Biobased Products and Bioenergy.

A biobased hydraulic fluid is currently defined as a fluid formulated with oils extracted from renewable resources such as plants, crops, trees or animals. The U.S. Department of Agriculture (USDA)'s biobased product guideline also defines exactly what products and how much concentration of renewable product associated with final product would be considered as a biobased product[3]. Currently, biobased hydraulic fluids are formulated with renewable products such as rapeseed, sunflower, corn, soybean, canola, and synthetic ester. These types of fluids are considered less toxic and more biodegradable that conventional hydraulic fluids[4]. The

Report Documentation Page

Form Approved
OMB No. 0704-0188

1. REPORT DATE **31 OCT 2007**	2. REPORT TYPE **N/A**	3. DATES COVERED **-**

4. TITLE AND SUBTITLE **Evaluation of Biobased Hydraulic Fluids in Military Construction Equipment**	5a. CONTRACT NUMBER 5b. GRANT NUMBER 5c. PROGRAM ELEMENT NUMBER
6. AUTHOR(S) **Rhee, In-Sik, Ph.D**	5d. PROJECT NUMBER 5e. TASK NUMBER 5f. WORK UNIT NUMBER
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) **USATACOM 6501 E 11 Mile Rd Warren, MI 48397-5000**	8. PERFORMING ORGANIZATION REPORT NUMBER **18436**
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)	10. SPONSOR/MONITOR'S ACRONYM(S) **TACOM TARDEC** 11. SPONSOR/MONITOR'S REPORT NUMBER(S) **18436**

12. DISTRIBUTION/AVAILABILITY STATEMENT
Approved for public release, distribution unlimited

13. SUPPLEMENTARY NOTES
To be presented the IFPE Hydraulic Fluid Symposium, Las Vegas, NV, 12-14 MAR 2008, The original document contains color images.

14. ABSTRACT

15. SUBJECT TERMS

16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT	18. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON
a. REPORT **unclassified**	b. ABSTRACT **unclassified**	c. THIS PAGE **unclassified**	**SAR**	**16**	

Standard Form 298 (Rev. 8-98)
Prescribed by ANSI Std Z39-18

chemical structures of vegetable oils are triglycerides in which a variety of saturated, monounsaturated or polyunsaturated fatty acids are esterified to a glycol backbone. The physical properties of a vegetable oil depend on the nature of its fatty acid composition. Some synthetic esters are also made from renewable sources instead of the petroleum sources. Their biodegradability is comparable to vegetable oils and the lubrication properties are very similar to mineral oils. But, they do not have identical chemical structures and lubrication properties[5].

In response to the demand of military BHFs, a limited field demonstration was conducted at Fort Bliss, TX, in 1999 using five experimental biodegradable hydraulic fluids (i.e., rapeseed oil, soybean oil, canola oil, synthetic ester oil) and ten construction and tactical equipment (i.e. scoop loaders, dump trucks, road graders, etc.)[6]. The field test results showed that all candidate BHF samples did not give any abnormal behavior and provided excellent service. Based on the successful completion of first Phase of this field demonstration, a new military performance specification, MIL-PRF-32073, Hydraulic fluid, Biobased, was developed to cover the hydraulic fluid requirements of military construction and tactical equipment[7] .

To introduce the biobased hydraulic fluids into military construction equipment, the second Phase of the field demonstration was initiated using the five BHFs qualified under MIL-PRF-32073 specification and ten pieces of construction equipment utilized in the Engineering School at Fort Leonard Wood, MO. This program was originally designed in a joint effort with USDA and Program Manager for Combat Engineer and Materials Handling Equipment (PM CE/MHE), and supported by Federal Biobased Products Preferred Procurement Program being developed to implement Section 9002 of the Farm Security and Rural Investment Act of 2002. The duration of this field test was one year and the acceptance of the BHFs in the construction equipment was based on the field testing evaluation and resultant finding generated. A construction equipment used in the demonstration is shown in Figure 1.

.

Figure 1. Bulldozer used in the Field Demonstration

FIELD DEMONSTRATION PROGRAM

The objective of this field demonstration was to verify performance of MIL-PRF-32073 Biobased Hydraulic Fluids (BHFs) for existing military construction equipment in cooperative effort with USDA, TARDEC Construction Engineer Team and Product Manager for Combat Engineer and Materials Handling Equipment (PM CE/MHE). Successful completion of this demonstration would result in the existing petroleum based hydraulic fluids used in construction equipment being replaced with non-toxic and biodegradable products.

The field test at Fort Leonard Wood, MO was focused on Biobased Hydraulic Fluids performance in military construction equipment and the potential environmentally acceptability of MIL-PRF-32073 fluids. The candidate BHF products were evaluated in a wide variety of military construction equipment. The duration of this field test was designed for a one year testing period. The final acceptance of the BHFs was based on the field testing evaluation and resultant findings generated. If MIL-PRF-32073 fluids are acceptable during this testing period, the field test will be extended to two more years to determine their service life.

Five BHFs qualified under the MIL-PRF-32073 specification were selected as field testing samples. These biobased fluids have been fully evaluated under laboratory environments, and have met all specification requirements. For the field test, 18 drums of MIL-PRF-32073 fluids were procured directly from four renewable oil companies. Their physical properties and identifications are described in Table 1. The fluids were identified by their codes.

The test samples were evaluated using the normal procedures utilized for construction equipment. A total of 10 pieces of construction equipment were used for this field test at Fort Leonard Wood. These vehicles belong to the Engineer School and are used for training Soldiers. The environment of this location has a typical Midwest weather and is a normal operation site. The test vehicles were selected based on availability and typical military applications and are listed in Table 2.

Table 1. Biobased Hydraulic Fluids Selected for the Field Demonstration

Code	Product Name	Viscosity @40 ºC	Pour point, ºC	Biodegradability, %
A	Cognis Proeco EAF 422LL	22.6	-51	66
B	Novus 100 ISO 46	42.0	44	72.7
C	Hydro Safe ISO VG68M5	68.6	37	68.3
D	Terresolve EL 146	46.6	25	85
E	Hydr Safe ISO32M3B	40.3	35	71

In preparation of this field demonstration, the existing petroleum based hydraulic fluids (SAE 15W-40 or MIL-PRF-2104) were completely removed from hydraulic systems of the construction equipment, and the inspection was conducted on the surface of these parts/components to determine whether the systems were leaking or not. The candidate BHFs were then introduced using the following changeover procedure;

(a) Operate the equipment for 15-20 minutes to warm the system.
(b) Drain the fluid from the reservoir and total systems such as pumps, lines and hoses.
(c) Refill the system with the appropriate fluid selected for each system and installs a new filter, and again operates the system for 15-20 minutes.
(d) At the end of the second warm-up period, drain and replace the fluid with a fresh change of a test fluid mentioned in the above step 3 and install a new filter.

After the completion of changeover procedure, the equipment was operated again for a short demonstration period to ensure that the hydraulic system is operated normally. The tested construction equipment must be operated a minimum for 5 hrs per week in routine military operations. During the tests all performance should be observed, and fluid level periodically inspected. The test fluids were sampled at the each quarter and their deterioration was evaluated in the laboratory.

Table 2. Construction Equipments Selected for Field Demonstration

Code	Name	Hour Operation, initial	Oil tank size, gal	Test oil
F-1	Bulldozer	4410	21	A
F-2	Bulldozer	3446	21	D
F-3	Scraper	2587	30	D
F-4	Scraper	2171	30	E
F-5	Grader	904	8	E
F-6	Grader	4050	8	A
F-7	Loader	3340	29	E
F-8	Excavator	1030	45	B
F-9	Crane	667	66	B
F-10	Crane	711	66	C

All testing results and operator/user comments were recorded. TARDEC collected data

including system inspections from maintenance personnel of Fort Leonard Wood and reviewed the data on a quarterly basis. The following performance characteristics were closely monitored at the testing site.

(a) Checked overall performance of new fluid and compared to existing conventional fluid.
(b) Any material incompatibility was observed (e.g., softens hoses, seals, etc).
(c) Low temperature operaterability was observed (e.g., fluid pumpability, freezing, etc.).
(d) Fluid evaporation in the system was checked (e.g., fluid level, etc.).
(e) Fluid condition was checked using laboratory tests (e.g., viscosity, TGA, PDSC, water content, etc.).
(f) Environmental assessment was determined (i.e., health and safety factors, operator acceptability, etc).

TEST RESULTS

A summary of the field demonstration is presented in Table 3. The equipment and fluids tested are identified by their designated codes. Data obtained for BHFs were generated from ten construction equipment that were utilized for the military training in Engineering School located at Fort Leonard Wood. Per the test plan, the tested equipment has been quarterly inspected and the field samples (2 OZ) were collected for the laboratory evaluation[8]. During the inspection periods, the equipment usages were recorded, and their hydraulic components and fluid levels were visually inspected (i.e., leaking spot, wear and corrosion problems, fluid condition, contamination, biodegradation, etc). Total equipment usages of this demonstration ranged from 50 to 393 hours operation. The usage of the construction equipment is normally measured by an hourly base rather than mileage of vehicles. Fort Leonard Wood is located at Midwest and its annual temperature ranges from 0 to 38 ºC. In this demonstration, any equipment or operational problems were not detected or notified from the 577th maintenance personnel and equipment operators. In addition, no biodegradation was observed in any equipment tested. In a visual inspection, the BHFs did not provide any abnormal behavior and have performed well in the construction equipment as the original petroleum based hydraulic fluids.

Hydraulic fluid is an essential and important component of any hydraulic power system. This fluid is currently formulated to provide the medium for efficient power transmission and lubrication to the system. In addition, the fluid should protect the system from corrosion and excessive wear, and must be compatible with seal materials to avoid leaking problems in the system. The excessive leaking of fluid can result in the loss of hydraulic power and create environmental problems such as soil contamination. In general, the fluid must be compatible with structural materials of the system and should exhibit stable physical properties during a suitable period of use and storage. Typically, the biobased fluid must not show any sign of biodegradation in the system.

Table 3. Results of Field Demonstration for Biobased Hydraulic Fluids

Code	Name	Equipment Usages (hrs)	Test oil	Leaking or other Operational problem	Fluid condition
F-1	Bulldozer	276	A	No	Clean
F-2	Bulldozer	258	D	No	Clean
F-3	Scraper	312	D	No	Clean
F-4	Scraper	286	E	No	Dark color
F-5	Grader	196	E	No	Clean
F-6	Grader	393	A	No	Clean
F-7	Loader	272	E	No	Clean
F-8	Excavator	50	B	No	Clean
F-9	Crane	135	B	No	Clean
F-10	Crane	243	C	No	Clean

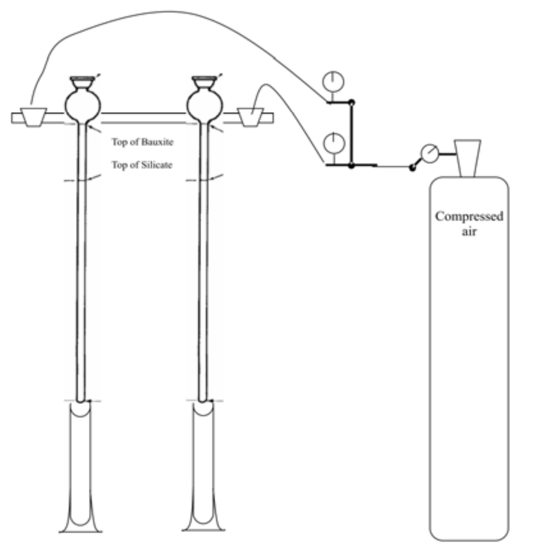
To evaluate the field samples, a test protocol was developed based on the above mentioned field performance criteria. It consists of viscosity testing, water content, oxidation stability, evaporation loss, low temperature stability, element analysis (wear), and composition analysis. Most of these tests

are currently specified in the MIL-PRF-32073 specification. Table 4 describes the test protocol used in this field demonstration. For the baseline study, the existing petroleum based hydraulic fluids (SAE 15W-40 or MIL-PRF-2104) collected from the hydraulic system of each piece of tested construction equipment were also evaluated according to the test protocol. Table 5 summarizes the test results of existing fluids by their equipment's code numbers. The composition analysis of the exiting fluids is presented in Table 6 with the biobased fluids. Table 7 -15 presents the laboratory test results of field samples collected at each quarter. The tested equipment and samples are represented by their designated codes.

Table 4. Test Protocol for Laboratory Evaluation

Test	Method
Viscosity	ASTM D 445
Water content	ASTM D 6304
Oxidation Stability	ASTM D 6186
Total Acid Number	ASTM D 664
Evaporation	ASTM E 1131
Low temperature Stability	ASTM D 6351
Elemental Analysis	X-ray Technique
Composition Analysis	ASTM D7373
Biodegradation	ASTM D 7373, ASTM D 6731

For the field demonstration, five BHFs were introduced into ten pieces of construction equipment. To increase the reliability of data, Fluids A, B, and D were each tested in two different pieces of equipment. Fluid E was tested in three pieces of equipment and Fluid C was tested in one construction equipment. According to the changeover procedure, all existing petroleum based fluids (SAE 15W-40 or MIL-PRF-2194) were completely drained and the hydraulic systems were refilled two times with tested biobased fluids to minimize the contamination from the existing hydraulic fluids. Then, the samples were collected from each equipment for the laboratory composition analysis. The composition analysis of the field samples are shown in Table 6 with their biobased fluids. The results showed that all biobased fluids were contaminated with 18 to 42.1 percent petroleum based fluids. It appeared that some of existing fluids were still remaining in the system due to the difficulty of cleaning the whole hydraulic system. For example, seals and hoses can become entrained with fluid which leaches out over time. Generally, it is almost impossible to remove all existing fluids from equipment except for a new system that is not lubricated with fluid. This is one issue raised when the different types of fluids are introduced into the existing system. Table 7 lists the actual composition of the tested fluid in each equipment. The composition test apparatus is



shown in Figure 2.

Figure 2. Composition Test Apparatus

Viscosity is an important property in hydraulic fluids and provides the lubrication of moving parts in a hydraulic system. This property directly affects flow characteristics, heat generation within system, pumping operation, sealing, leaking characteristics. The viscosity of fluids is often measured using the ASTM D 445, Kinetic Viscosity of Transparent and Opaque Liquids. This test method covers the determination of kinetic viscosity of fluids. The viscosity values most frequently measured for a fluid are at 40 and 100 ºC at atmospheric pressure and low-shear rates. Currently, military biobased hydraulic systems use many

different types of viscosity grades (ISO VG 15, 22, 32, 46, and 68) as an operational fluid. For this reason, the five different types of viscosity grades were selected for the test samples. Viscosity index (VI) is also used for measure of how viscosity changes with temperatures. Generally, a high VI indicates that the viscosity of fluids undergo less change with temperature variations. Table 8 summarizes the viscosity data of all samples collected at each vehicle inspection period. The viscosity data of biobased samples collected from original drums are also presented in Table 8. They usually change slightly with time due to the test precision and aging. It was observed that the viscosities of field samples were not significantly changed over the one year period. Also, the viscosities of biobased fluids obtained from drums were not significantly changed for a year. This result indicated that the field samples were still in good condition.

During the vehicle inspection period, it was noted that the operational temperature of hydraulic system (about 37.8 ºC) tended to increase 5 ºC in some equipment (i.e., Crane), but it did not affect the operation of the hydraulic system. It appeared that some biobased fluids have a lower thermal stability than that of the petroleum based fluids. The viscosity of samples collected at third quarter from a Grader (F-6) showed a very significant viscosity change. This data was considered an outlier. It is possible that the equipment operator or mechanics might have accidentally topped off the system with the petroleum based fluid (SAE 15W-40) instead of BHF. However, this contamination problem was not observed anymore during the fourth quarter. It appears that the significant amount of top off fluid may change the viscosity of existing fluid in the system. Overall, no significant viscosity changes were observed in any equipment used in this demonstration.

The oxidation stability is the ability of fluids to resist oxidation at elevated temperatures. This property is another important operational parameter in military hydraulic systems and directly affects fluid service and storage life. Most hydraulic fluids contain some degree of the oxidation inhibitors to reduce the oxidation process during service. In a visual inspection, if a fluid is oxidized, it is usually demonstrated by a darkening in color and the change in viscosity. In addition, the fluid may be decomposed and polymerized in the system. Eventually, this property can lead to degraded service life. Several laboratory tests are available to measure this property. Currently, the ASTM D 664, Acid Number of Petroleum Products by Potentiometric Titration, is widely used to measure the oxidation stability of fluid. In this test, Acid Number (typically referred to as TAN) is the most common measure of fluid acidity and represents its degree of degradation. Generally, increasing TAN over time indicates deterioration of the fluid. Table 9 presents the test results of TAN obtained from the field samples.

The test results showed that the TANs of fluids were very stable over times and marked low values. Some of fluids tended to decrease their TANs over time rather than increase. Based on these data, it is difficult to make a judgment for the oxidation or deterioration of the fluids because there was no other indication of oxidation or deterioration in the field samples.

To verify these results, another oxidation test was conducted using the ASTM D 6186, Oxidation Induction Time of Lubricating Oils by Pressure Differential Scanning Calorimetry (PDSC). This method is also widely used to measure the oxidation stability of fluids under oxygen environments. In this test, the degree of oxidation stability at a given test temperature is determined by an induction time. One benefit of this test is that it is used to calculate the oxidation of field samples using an oxidation kinetic model[9].

Table 10 summarizes the PDSC test results obtained at 180 ºC. Sample F-2 does not show consistent results at each quarter. In this PDSC test, the induction times of fluids always decrease with time due to the oxidation of fluid. Therefore, it is suspected that the data obtained from F-2, except for the first quarter, may be the resulted accidental top off with the other types of biobased fluids or the existing petroleum based fluids. Generally, all samples showed some degree

of oxidation in the equipment and storage. This is considered a part of the fluid aging process. It was reported that the field samples were oxidized range from up to 53.8 % for this testing period. If a fluid was oxidized more than 90 % in this test, its useful life is over and it requires an oil change with new fluid. The data obtained from original drums showed very low oxidation occurred in the fluids.

All fluids tested remained in good condition and there was no major degradation during this demonstration. Fluid B tested in an Excavator (F-8) had a higher oxidation (53.8 %) than the others. This result agreed somewhat with the viscosity test, but it did not directly agree with the acidity test. It appeared that the additives used in Fluid B may have depleted during this period and fluid became increasingly more acidic. Generally, the increasing in TAN of fluid indicates the depletion of the oxidation inhibitor utilized in fluid. In fact, the reduction of oxidation inhibitor in fluid tends to increase its oxidation rate.

Low temperature properties of BHFs are important, particularly when storing fluids in cold environments or when hydraulic systems are subjected to periods of nonoperation in cold environments. Formation of gels or crystals or separation of components can cause clogging of filters, plugging of small orifices and clearances thus resulting in lack of lubrication to vital components. The freezing of fluid in the reservoir will create pumping problems in the field. The low temperature properties of fluids are directly related to their viscosity grades. A low viscosity grade provides a better low temperature performance in a low temperature environment. The field samples classified in ISO VG 22, 46, and 68 have different low temperature properties in cold environments. Their low temperature properties were measured using the ASTM D 6351, Determination of Low Temperature Fluidity and Appearance of Hydraulic Fluids. Table 11 presents their low temperature characteristics. There was no change of low temperature stability before or after the field demonstration. In addition, none of fluids failed in the field and their low temperature performance was the same as those of the existing petroleum based fluids. It is noted that the lowest temperature during the field testing at Fort Leonard Wood was around 0 ºC.

Hydrolytic stability is the ability of hydraulic fluids to resist reaction with water. Even though the hydraulic system is well sealed, the moisture is difficult to exclude because temperature changes cause reservoir breathing and condensation of moisture from environments. Some of ester based fluids can absorb moisture from the environment. Then, the ester is hydrolyzed to an acid and alcohol. For this reason, the high water content in fluids may affect fluid life, and cause corrosion and biodegradation problems in hydraulic systems. Typically, the BHFs tend to have a low hydrolytic stability in compared to the petroleum based fluids. Because of this, fluid samples were monitored at each quarter for water content. Table 12 presents the test results obtained from the ASTM D 6304, Determination of Water in Petroleum Products, Lubricating Oils, and Additives by Coulometric Karl Fisher Titration. The test results showed that field samples did not generate or absorb water.

Volatility is the rate at which a fluid will vaporize. The hydraulic fluid when exposed to high temperatures at atmospheric pressure can result in significant loss of fluid, and tends to increase in both viscosity and density. In addition, highly volatility in the field samples is more likely to indicate the oxidation of fluid, and to lead to cavitations and hydraulic pump damage. Generally, BHFs do not have a volatility problem at the operating temperature of hydraulic systems (about 50 ºC). To verify this property in the field, the evaporation test (ASTM E 1131) was conducted on the field samples. Table 13 summarizes the TGA test results obtained at 100 ºC for an hour. It appeared that BHFs did not have a volatility problem in this demonstration.

Seals sometimes fail to perform their designed function of retaining hydraulic fluids and excluding contaminants because of incompatibility between the seal elastomer and the hydraulic fluid. The deterioration of elastomer seals results in the failure of

hydraulic power and cause leaking of fluids. Numerous seal materials are currently used in the hydraulic systems. They are basically divided into two groups. One is natural rubber and the other one is a synthetic elastomer. Their formulations and physical properties are not same. Such materials are usually modified by additives, fillers, and other ingredients and then cured to make a finished elastomer compound. The number of basic elastomers is small, but the variety of finished compounds that can be made is almost infinite. The hydraulic fluid usually has more effect on the seal or packing compound than the compound has on the hydraulic fluid. In most instances the compound is almost inert as far as any harmful effects on the fluids are concerned unless additives in the seal materials are extracted by the fluid. However, some fluids can attack and destroy seal materials.

The best method of determining whether a fluid and an elastomer are compatible is to observe the use in actual hydraulic systems. Unfortunately, this approach is almost impossible in the practical sense due to the variation of seal materials being used in the equipment. For this reason, most of hydraulic fluid specifications often require a reference elastomer for the elastomer compatibility test. The biobased hydraulic fluid specification also uses SAE reference elastomer (AMS 3217/2B) with its specification limit for the seal compatibility test. This reference elastomer was specially formulated with Nitrile or Buna N (NBR-L) material and is widely used in the many hydraulic specifications including military specifications. The swelling limits (10 to 30 percent) used in this specification were developed based on the previous field demonstration for biobased hydraulic fluids.

Prior to the field demonstration, all samples were evaluated according to the laboratory elastomer compatibility test method and had passed its specification requirement. During the field demonstration, none of the equipment had seal material failures nor had a leaking problem in any part of their components.

Metal compatibility is a very important property in a hydraulic fluid. The copper, silver, bronze, aluminum, steel, iron and many other metals are commonly used as structural materials in hydraulic systems. Normally, the corrosion or rust on metal surface is one of indications for the incompatibility of fluids. The fluid compatibility with metals can be measured by a number of tests. These techniques usually involve exposing the metal to the fluid under a variety of conditions and determining any changes in the fluid or the metals. The biobased hydraulic fluid specification requires three different types of corrosion tests to evaluate the compatibility between fluids and metals: copper corrosion test, galvanic corrosion test, and rust prevention test in synthetic seawater. These corrosion tests were originally designed to evaluate specific corrosion characteristics of fluids in different applications and environments. One example is the galvanic corrosion test that is designed to determine the fluid–metal compatibility between dissimilar metals during use. Like the conventional petroleum based hydraulic fluids, the biobased fluids must be compatible with all common metals used in construction of hydraulic systems. To verify this property in the field, all field samples including existing petroleum based fluids were analyzed using an X-ray technique.

Table 14 summarizes the results of element analysis for all samples. In this element analysis, 12 chemical materials including five metals (Fe, Ni, Mg, Cu, and Zn) were analyzed. Unlike the petroleum based fluids, the BHFs evaluated did not contain organo-metal additives. However, field samples contaminated with petroleum based fluid showed Zinc metal in this analysis. This metal was also found in the engine oils and it appeared that this element came from the ZDDP anti-wear additive utilized in the MIL-PRF-2104 petroleum based fluids. The BHFs did not show any evidence of incompatibility between BHFs and structural materials in hydraulic systems.

Environmental compatibility of hydraulic fluids is a very important parameter today. A common problem in most hydraulic systems is the potential for leakage and the possibility of hydraulic fluid spilling during use. The generation of hazardous wastes from fluids

results in both short- and long- term liability in terms of cost, environmental damage, and mission performance. To resolve this problem, the biobased hydraulic fluids are currently used in environmentally sensitive areas such as construction, forestry, mining, and river. The major benefits of biobased fluids are low toxicity and high biodegradability. In addition, they are non-carcinogenic, and do not contain any prohibited ingredients listed by EPCRA, CERCLA, and RCRA.

During the field demonstration, the BHFs were handled by a normal maintenance procedure and did not give any skin and eye irritation on maintenance people. One of questions raised in this study was whether the field samples still can provide a high biodegradability when compared with their original fluids. Any incompatibility between fluids may reduce their biodegradability due to the chemical reaction. Generally, the biodegradability of the fluid depends on its material and chemical structure. It was observed that the BHFs did not biodegrade in the hydraulic systems (i.e., reservoirs) during the field demonstration.

To determine the biodegradability of fluids, the field samples were analyzed to predict their biodegradability according to the ASTM D 7373, Predicting Biodegradability of Lubricants using a Bio-kinetic Model. Table 6 summarizes the composition analysis for field samples and their predicted biodegradability is presented in Table 15. To verify these results, the actual biodegradation tests were also conducted using fourth quarter samples according to the ASTM D 6731, Determining the Aerobic, Aquatic Biodegradability of Lubricants or Lubricant Components in a Closed Respirometer. These test results are also presented in Table 15. The test results showed that all field samples did not change their biodegradability in the systems and storages (drums) over time. The field samples containing petroleum fluid had a relatively lower biodegradability than BHFs because the petroleum based fluids generally are considered non-biodegradable products. There was no indication of chemical degradation in this composition analysis.

In the changeover or adding hydraulic fluids, one question was the compatibility of biobased fluid versus the existing petroleum based fluid in the construction equipment. Incompatibility of fluids is usually evident in their physical and chemical properties. Typically, it shows in the viscosity tests and evaporation loss, composition analysis, or seal compatibility test due to their internal chemical reaction. As mentioned earlier, all samples were already contaminated with the existing petroleum based fluids (16.8 to 42.1 %). During the field demonstration, no sign of incompatibility between two fluids was observed and all laboratory data supported this result. It implied that the biobased fluid can easily be changed over from the existing petroleum based fluid without any major cleaning effort to the system.

CONCLUSIONS

Field demonstration to evaluate the performance of biobased hydraulic fluids in pieces of construction equipment was successfully completed. The tested biobased fluids did not show any abnormal behavior in this demonstration and provided performance equivalent to the existing petroleum based fluids. In addition, no equipment failed or was damaged due to the biobased fluids. During this period, neither biodegradation nor chemical degradation was observed in hydraulic fluid systems and reservoirs. One concern in this demonstration was the low temperature stability and operatability of biobased fluids in the field environment. The tested fluids, except for Fluid A, were not formulated for use at extreme low temperatures (i.e., -40 to -54 ºC). In this demonstration, it was observed that fluids did not have low temperature operational problem due to the milder Midwest winter weather.

The physical and chemical properties of fluids were slightly changed due to the aging of the fluids. This is considered a normal degradation process of any fluid. Typically, the biodegradability of the tested fluids did not change during the field demonstration. In addition, the biobased fluids did not show any incompatibility with the existing petroleum

based fluids and the seal materials used in construction equipment. It appears that the biobased fluid can easily be changed over from the existing fluids without any major cleaning effort to the hydraulic system. A small amount of petroleum based hydraulic fluid does not create any incompatibility or operational problems in the system. In addition, the field demonstration results were found to be in good agreement with the laboratory performance test used in this study.

All field demonstrations were successfully completed, met original milestones, and did not impact the military mission of the unit in any way. The overall performance of BHFs have been proved in the field demonstration and accepted by military users. Therefore, the BHFs can be used as an operational fluid for military construction equipment. The following recommendations were made based on this study.

(1)Select biobased fluids meeting MIL-PRF-32073 as military operational fluids for construction equipment.
(2) Conduct extended field demonstration to determine extend of service life for biobased fluids in construction equipment.
(3) Consider a study to convert waste biobased fluid to a biofuel.

Acknowledgments

This field demonstration program was supported by Office of Energy Policy and New Uses, USDA and Program Manager for Combat Engineer and Materials Handling Equipment (PM CE/MHE), Authors thank Ms. Camela Bailey of USDA for helping and supporting this program and Mr. Michael Parsons and Mr. Jeffrey Anderlik at Fort Leonard Wood for actually performing this demonstration. Authors also wish to thank those who provided biobased hydraulic fluids for this demonstration.

REFERENCES

1.Klaus Pelinka, “Environmental Restoration Effort Takes on Momentum”, National Defense, October, 1993.
2.The Resource Conservation and Recovery Act of 1976, 42 U.S.C. 6901 et seq., 1976; the Clean Water Act of 1972 and amended in 1987.
3. Guidelines for Designating Biobased Products for Federal Procurement, Federal Resister, Volume 71, Number 51, USDA, March 16, 2006.
4.In-Sik Rhee, “Development of Military Biodegradable Hydraulic Fluids”, SAE 2002 01-1503, SAE, Warrendale, PA, 2002.
5.In-Sik Rhee, “Evaluation of Environmentally Acceptable Hydraulic Fluids”, NLGI Spokesman, 1996.
6.B.R. Wright, R.A. Alvarez, G. Phillips, and In-Sik Rhee, “Field Demonstration of Biodegradable Hydraulic Fluid in Military Tactical and Construction Equipment”, SwRI Interim Report, TFLRF No. 339, 1999.
7.Military Specification MIL-PRF-32073A, Hydraulic Fluid, Biobased, 2007.
8. In-Sik Rhee, Trip Reports for Biobased Hydraulic Fluids Field Demonstration, 2005-2006.
9.In-Sik Rhee, “Decomposition Kinetics of Greases by Thermal Analysis, 74th NLGI Annual Meeting, June, 2007.

Table 5. Test Results obtained from Existing Petroleum based Hydraulic Fluids Collected from Tested Equipment

Code	Viscosity (cSt)			Oxidation Stability @180ºC, min	Evaporation @100 ºC for 1 hr, %	Total Acid number Mg KOH/g	Low temperature Stability (minimum temperature, ºC)
	40 ºC	100 ºC	VI*				
F-1	81.97	10.88	119.5	24.03	0.7516	2.30	-25
F-2	69.91	9.904	123.6	40.22	0.6497	2.59	-25
F-3	53.11	8.147	124.1	90.96	0.5353	2.11	-25
F-4	64.04	9.486	128.6	76.70	0.390	2.15	-25
F-5	60.10	8.596	116.0	31.36	1.335	2.01	-25
F-6	55.50	8.288	122.2	21.83	1.829	1.87	-25
F-7	35.76	6.133	118.9	58.16	0.8824	1.65	-25
F-8	39.59	6.689	124.5	36.26	0.8058	1.97	-25
F-9	44.41	7.104	119.7	30.99	1.029	2.24	-25
F-10	44.06	7.083	120.5	23.48	1.048	2.15	-25
SAE 15W-40	97.56	13.60	142.2	14.0 @210C	0.3636	2.55	-25
MIL-PRF-2104	45.8	12.8		53.37	0.9006	2.27	-25

Table 6. Composition Analysis for Field Samples

Sample		Chemical Composition, %							
Code	I.D.	Saturate		Non-polar Aromatics		Ester		Polar Aromatics	
		1Q	4Q	1Q	4Q	1Q	4Q	1Q	4Q
F-1	A	55.49	53.61	3.99	3.91	33.76	39.63	6.76	2.85
F-2	D	14.21	14.97	4.94	3.89	74.07	76.94	6.78	4.20
F-3	D	26.02	28.33	5.22	5.86	61.09	60.80	7.68	5.0
F-4	E	34.21	34.05	6.15	4.73	53.03	59.4	6.61	1.82
F-5	E	42.96	52.12	9.17	8.32	41.26	36.58	6.61	2.98
F-6	A	61.35	61.47	7.61	7.06	21.13	23.47	9.91	8.0
F-7	E	39.77	40.44	5.11	4.97	46.99	48.34	8.14	6.26
F-8	B	33.24	26.39	10.55	5.91	53.89	66.66	2.32	0.54
F-9	B	18.43	27.00	4.30	5.59	73.29	72.24	3.98	6.03
F-10	C	35.77	31.84	4.05	3.89	58.06	58.4	2.14	5.84
A	-	50.50	50.13	0.61	1.20	39.32	39.63	9.57	9.03
B	-	3.30	3.29	3.01	3.13	90.52	87.3	3.16	6.27
C	-	15.76	15.65	2.56	2.20	75.99	78.91	5.69	3.24
D	-	2.65	2.52	2,14	2.63	89.54	88.42	5.67	6.44
E	-	16.22	14.74	1.56	2.44	76.66	77.81	5.56	5.0
SAE 15W-40	-	86.59	-	10.69	-	1.48	-	1.32	-

MIL-PRF-2104	-	77.48	-	19.94	-	1.25	-	1.34	-

Table 7. Composition of the Tested Samples in the Equipment

Equipment Code	SAE 15W-40 or MIL-PRF-2104, %	Biobased Fluid from MIL-PRF-32073, %
F-1	19.7	80.3
F-2	16.8	83.2
F-3	34	66
F-4	18	82
F-5	30.7	69.3
F-6	40.9	59.1
F-7	23.6	76.4
F-8	42.1	57.9
F-9	18.1	81.9
F-10	28.8	71.2

Table 8. Viscosity Test Results

Sample Code	1Q			2Q			3Q			4Q		
	40˚C	100˚C	VI	40˚C	100˚C	VI	40˚C	100˚C	VI	40˚C	100˚C	VI
F-1	25.75	5.403	**151.4**	25.5	5.37	**151.5**	25.35	5.323	**149.8**	26.18	5.470	**152.0**
F-2	45.72	9.971	**212.8**	57.73	11.70	**203.1**	57.43	11.44	**198.1**	57.50	11.41	**197.1**
F-3	42.63	8.966	**198.3**	42.26	8.751	**192.8**	42.13	8.793	**195.0**	42.13	8.73	**192.7**
F-4	42.95	8.417	**177.0**	43.3	8.372	**173.3**	42.94	8.319	**173.3**	44.90	8.74	**177.9**
F-5	45.56	8.337	**160.8**	37.90	7.218	**157.3**	37.98	7.204	**156.2**	41.02	7.562	**154.2**
F-6	32.19	6.148	**142.5**	31.4	6.001	**140.2**	83.31	11.30	**126.7**	31.08	5.954	**140.0**
F-7	38.07	7.544	**170.5**	32.02	6.557	**165.5**	31.72	6.492	**164.3**	31.56	6.461	**163.7**
F-8	38.70	7.837	**179.7**	39.25	7.949	**180.2**	41.77	8.66	**192.4**	41.69	8.646	**192.3**
F-9	42.32	8.713	**191.0**	42.81	8.551	**182.4**	41.6	8.462	**186.1**	41.29	8.415	**186.2**
F-10	57.18	10.68	**180.4**	58.39	10.76	**177.9**	59.54	10.70	**172.3**	56.43	10.46	**177.6**
A	23.64	5.184	**157.9**	-	-	**-**	-	-	**-**	22.39	4.872	**146.3**
B	42.11	9.069	**204.8**	-	-	**-**	-	-	**-**	43.34	9.215	**202.4**
C	67.65	12.70	**190.7**	-	-	**-**	-	-	**-**	68.06	12.73	**190.1**
D	49.18	11.03	**224.7**	-	-	**-**	-	-	**-**	48.60	10.99	**226.5**
E	40.70	8.409	**189.5**	-	-	**-**	-	-	**-**	40.88	8.395	**187.9**

Table 9. Test Results of Total Acid Number

Sample Code	1Q	2Q	3Q	4Q
F-1	0.40	0.35	0.37	0.41
F-2	0.24	1.1	0.91	0.42
F-3	0.62	0.42	0.45	0.42
F-4	1.29	0.88	0.88	0.40
F-5	1.62	0.79	0.89	0.64

F-6	0.70	0.77	1.95	0.81
F-7	0.36	0.63	0.63	0.23
F-8	0.90	0.69	0.56	0.45
F-9	0.45	0.64	0.58	0.48
F-10	1.02	1.03	1.34	0.88
A	0.21			0.23
B	0.94			1.07
C	1.06			0.83
D	0.33			0.10
E	1.67			1.52

Unit: mg KOH/g

Table 10. PDSC Test Results (minutes) at 180 ºC

Sample Code	New Fluid	1Q	2Q	3Q	4Q	% Oxidation
F-1	36.06 (A)	24.87	29.25	32.04	18.5	8.4
F-2	2.88 (D)	2.0	9.03	7.55	7.48	9.8**
F-3	4.14 (D*)	3.07	3.49	4.64	3.79	0.5
F-4	39.3 (E)	39.33	40.65	38.54	27.9	2.8
F-5	39.31 (E)	14.66	15.55	21.7	17.3	12.2
F-6	36.06 (A)	19.47	18.26	65.8	17.41	9.9
F-7	39.3 (E)	32.05	41.44	43.68	40.1	0.01
F-8	4.53 (B)	3.87	2.62	2.73	1.02	53.8
F-9	4.53 (B)	2.4	2.62	2.84	2.15	27.1
F-10	19.7 (C)	6.39	5.66	5.98	6.01	23.6
A	36.06	-	-	-	36.1	6.8
B	7.8	-	-	-	7.7	7.7
C	19.7	-	-	-	19.4	0.08
D	2.88	-	-	-	2.29	5.7
D*	4.14	-	-	-	4.14	0
E	39.3	-	-	-	26.74	2.4

* Different Batch of Fluid ** calculated based on 1Q data

Table 11. Low Temperature Stability Test (ºC)

Sample Code	1Q			2Q			3Q			4Q		
	-15	-25	-40	-15	-25	-40	-15	-25	-40	-15	-25	-40
F-1	Flow	Flow	Flow	Flow	Flow	Flow	Flow	Flow	Flow	Flow	Flow	Flow
F-2	Flow	Flow	Frozen	Flow	Flow	Frozen	Flow	Flow	Frozen	Flow	Flow	Frozen
F-3	Flow	Flow	Frozen	Flow	Flow	Frozen	Flow	Flow	Frozen	Flow	Flow	Frozen
F-4	Flow	Flow	Frozen	Flow	Flow	Frozen	Flow	Flow	Frozen	Flow	Flow	Frozen
F-5	Flow	Flow	Frozen	Flow	Flow	Frozen	Flow	Flow	Frozen	Flow	Flow	Frozen
F-6	Flow	Flow	Flow	Flow	Flow	Flow	Flow	flow	flow	Flow	Flow	Flow
F-7	Flow	Flow	Frozen	Flow	Flow	Frozen	Flow	Flow	Frozen	Flow	Flow	Frozen
F-8	Flow	Flow	Frozen	Flow	Flow	Frozen	Flow	Frozen	Frozen	Flow	Frozen	Frozen
F-9	Flow	Flow	Frozen	Flow	Frozen	Frozen	Flow	Frozen	Frozen	Flow	Flow	Frozen
F-10	Flow	Flow	Frozen	Flow	Flow	Frozen	Flow	Flow	Frozen	Flow	Flow	Frozen
A	Flow	Flow	Flow	-	-	-	-	-	-	Flow	Flow	Flow
B	Flow	Flow	Frozen	-	-	-	-	-	-	Flow	Flow	Frozen
C	Flow	Flow	Frozen	-	-	-	-	-	-	Flow	Flow	Frozen
D	Flow	Flow	Frozen	-	-	-	-	-	-	Flow	Flow	Frozen
E	Flow	Flow	Frozen	-	-	-	-	-	-	Flow	Flow	Frozen
SAE 15W-40	Flow	Gelling	Frozen	-	-	-	-	-	-	-	-	-
MIL-PRF-2104	Flow	Slow flow	Frozen	-	-	-	-	-	-	-	-	-

Table 12. Total Water Content, %

Sample Code	I.D.	1Q	2Q	3Q	4Q
F-1	A	.028	.027	.095	.079
F-2	D	.027	.263	.129	.083
F-3	D	.042	.097	.142	.117
F-4	E	.76	.137	.268	.108
F-5	E	.64	.158	.252	.184
F-6	A	.106	.345	.592	.271
F-7	E	.065	.146	.141	.103
F-8	B	.069	.155	.127	.077
F-9	B	.074	.217	.298	.201
F-10	C	.094	.137	.205	.183
A	Drum	.042	-	-	.042
B	Drum	.052	-	-	.087
C	Drum	.061	-	-	.077
D	Drum	.050	-	-	.055
E	Drum	.068	-	-	.142
SAE 15W-40	Drum	.315	-	-	-
MIL-PRF-2104	Drum	.267	-	-	-

Table 13. TGA Test Results (%) at 100 ºC, 1 hr

Sample Code	New Fluid	1Q	2Q	3Q	4Q
F-1	0.813 (A)	1.896	0.989	1.716	1.675
F-2	1.508 (D)	1.812	0.933	0.6963	0.660
F-3	1.508 (D)	1.288	1.898	1.676	1.328
F-4	0.325 (E)	0.457	0.542	0.4623	0.438
F-5	0.325 (E)	1.12	0.972	2.696	1.038
F-6	0.813 (A)	1.21	2.76	0.5750	1.235
F-7	0.325 (E)	0.662	1.196	1.417	0.829
F-8	0.842 (B)	0.981	0.961	0.9822	0.924
F-9	0.842 (B)	1.11	1.136	0.8971	0.910
F-10	0.218 (C)	0.981	0.554	0.4916	0.436
A	0.813	-	-	-	1.387
B	0.842	-	-	-	0.798
C	0.218	-	-	-	0.173
D	1.508	-	-	-	1.4
E	0.325	-	-	-	0.358

Table 14. Elemental Analysis by X-ray Method

Sample ID	Concentration – ppm											
	Mg	**Si**	**P**	**S**	**Cl**	**Ca**	**Cr**	**Mn**	**Fe**	**Ni**	**Cu**	**Zn**
A	0	0	380	667	0	0	0	0	0	0	1	0
B	0	0	0	0	45	711	0	0	0	0	0	0
C	0	0	193	856	0	13	4	0	0	0	0	0
D	0	0	528	1166	0	14	0	0	0	0	0	0
E	0	0	189	823	0	13	0	0	1	0	2	0
SAE 15W-40	274	0	1303	4469	126	2174	0	0	3	0	0	1338
MIL-PRF-2104	0	27	1098	7000	157	2547	0	0	2	1	0	1140
F1-4	0	0	471	1535	0	243	0	0	0	0	6	147
F2-4	0	0	324	1200	0	74	0	0	0	0	2	54
F3-4	81	0	685	2247	0	545	4	0	0	0	0	325
F4-4	30	0	369	1883	0	407	0	0	2	1	4	239
F5-4	210	0	559	2705	140	565	0	0	3	0	24	447
F6-4	214	13	590	2514	102	454	0	0	4	1	122	388
F7-4	0	0	364	1586	72	269	0	0	5	0	0	171
F8-4	36	0	241	1904	0	294	0	0	3	0	58	232
F9-4	154	0	298	1895	0	284	0	0	0	0	40	276
F10-4	156	13	365	1683	0	228	0	0	0	1	48	238

Table 15. Biodegradability of Field Samples

Sample Code	Tested Sample Composition	ASTM D 7373		ASTM D 6731
		1Q (%)	4Q (%)	4Q (%)
F-1	A (80.3%) +P* (19.7%)	64	68	60.8
F-2	D (83.2%) +P (16.8 %)	66	68.8	74.8
F-3	D (66 %) + P (34 %)	59	60	67.3
F-4	E (82 %) + P (18 %)	55.4	59.8	57
F-5	E (69.3 %) + P (30.7 %)	49.4	48	ND
F-6	A (59.1 %) + P (40.9 %)	41	42	61.7*
F-7	E (76.4 %) + P (23.6 %)	52.4	53.4	69
F-8	B (57.9 %) + P (42.1 %)	55.7	63.4	64.4
F-9	B (81.9 %) + P (18.1 %)	66.7	67.9	71.3
F-10	C (71.2 %) + P (28.8 %)	59.2	58.6	74.1*
A from Drum	100% Bio-based Fluid	66	66	66
B from Drum	100% Bio-based Fluid	76.7	74	72.7
C from Drum	100% Bio-based Fluid	67	69.7	68.3
D from Drum	100% Bio-based Fluid	75.6	75	85
E from Drum	100% Bio-based Fluid	68	68.5	71
SAE 15W-40 from Drum	100% Petroleum Fluid	33.7	-	34.1

MIL-PRF-2104 from Drum	100% Petroleum Fluid	22.6	-	30.0

(1) Petroleum based existing fluid (SAE 15W-40 or MIL-PRF-2104).
* Considered as an outlier due to the test equipment problems.